Green Synthesis and Characterization of Zinc Oxide and Iron Oxide Core Shell Nanostructure

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By

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INTRODUCTION

With the advent of nanotechnology, many nano-scale devices have been developed using numerous methods, such as physical, chemical, and green approaches. Yet, green nanoparticle synthesis is a tool of choice that can be easily prepared and engineered and are less expensive. Green synthesis of metal oxide nanoparticles using microorganisms, enzymes, and plants [Ocimum tenuiflorum(Tulsi)] or plant extracts has been suggested as a possible eco-friendly alternative to chemical and physical methods. Nanotechnology also plays an interesting role in many areas of computing, power generation, optics, drug delivery, and environmental sciences. Due to their multiple applications in various technical and medical fields, comprehensive investigation into metal oxide nanoparticles has been concentrated in the past decade.

The objectives of this present study are the biosynthesis of iron oxide and zinc oxide nanoparticles, they received a lot of attention from researchers, due to their potential properties such as optical, magnetic, electronic, and chemical properties and hence have incredible applications in the field of physics, agriculture, food processing, chemistry, medicine, biology and so on. ZnO and Fe_2O_3 -NPs can be used in various sectors, such as energy conservation, textiles, electronics, healthcare, catalysis, cosmetics, semiconductors, and chemical sensing. However. The Conventional methods are long-term processing, laborious

procedures, and in particular, the use of toxic compounds and are energy consuming, using hazardous solvents and expensive reagents in their preparations. Different kinds of methods have been reported for the synthesis of zinc oxide and iron oxide nano-particles such as thermal decomposition methods, coprecipitation techniques, hydrothermal techniques, sol-gel methods, electrochemical methods, etc. Most of the relevant study has been directed to eco-friendly, safer, eco-sustainable, and fast synthesis protocols for the production of nanoparticles due to these limitations, Therefore the a rising need to develop an eco-friendly and safer green method for nanoparticle preparation. In this respect, green synthesis of ZnO and Fe_2O_3 NPs, especially using extracts from different kinds of plants, is a growing trend that is considered simple, cheap, eco-friendly, and non-toxic. [1]In recent years, zinc oxide (ZnO), an important semiconductor (Iron oxide also) with tremendous scientific and technological interest, having a direct wide gap (3.37 eV, 387 nm, deep violet/ borderline ultraviolet (UV)) and large exciton-binding energy (60meV), is a highly preferred multitasking metal oxide having a vast list of attractive properties. Due to its unique electrical and optical properties.[2]

Recently plant-mediated green synthesis of different metal oxide nanoparticles from various plants has been reported such as Cassia alata and Mangifera indica. ZnO and Fe_2O_3 nanoparticles have been widely used in many industrial areas such as solar cells, UV light-emitting devices, Cosmetics, gas

sensors, photocatalysts, pharmaceuticals, and many more industries. Additionally, metal oxide nanoparticles have a surface plasmon resonance absorption(PRA) in the UV–visible region. It is non-toxic, self-cleansing, compatible with the skin, antimicrobial, and dermatological and is used as a UV-blocker in sunscreens and many biomedical applications. The green synthesized magnetic oxide nanoparticles will be characterized using modern techniques/methods such as ultraviolet (UV) spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM), dynamic light scattering (DLS), transmission electron microscopy (TEM), and VSM. The nanoparticles will be checked for their antimicrobial, antileishmanial, antidiabetic, antioxidant, antilarvicidal, and protein kinase inhibitory potential. . More research on green synthesis of zinc oxide and iron oxide nanoparticles is needed to explore their applications in biomedicine both in vitro and in vivo levels.

OBJECTIVES

The following objectives have been proposed

1. To develop a green solution-based synthesis technique for the growth of ferrite Iron oxide – Zinc Oxide nanoparticles by using Ocimum tenuiflorum(Tulsi).
2. To investigate the structural, morphological, optical, and magnetic properties.
3. To examine a possible correlation between the growth process variables and the observed properties.

REVIEW OF LITERATURE

Over the past few years, spinel ferrite nanoparticles have attracted considerable attention. Ferrites are among the most widely used electromagnetic materials for a broad category of applications over a wide frequency range due to their low cost and high performance. Recently, ferrite research has been shifted towards developing these materials in nanometric scales as the performance in their conventional bulk preparation routes is reaching its limits due to their domain wall resonance and higher electrical conductivity. However, technological

advances in the electronics industry demand even more compact cores for work at higher frequencies. One way to solve this problem is by synthesizing the ferrite particles in nano scales before compacting them for sintering by using Ocimum tenuiflorum(Tulsi). When the size of the magnetic particle is smaller than the critical size for multidomain formation, the particle exists in a single domain state, and domain wall resonance is avoided; thus the material can work at higher frequencies.

Due to the enormous applications of nanoparticles, the demand for their synthesis rises despite persisting difficulties in larger-scale production. Several methods such as sol-gel, hot spraying, microemulsion method, co-precipitation, rf plasma torch method, chemical vapor deposition, solvothermal, and combustion methods are used for the synthesis of these materials from Ocimum tenuiflorum(Tulsi). Out of these the chemical co-precipitation method is found to be appropriate for the synthesis of uniform particles at near room temperature[3].

Some methods of preparation of ferrites nanoparticles are:-

1. Chemical Co-precipitation method:

Co-precipitation is one of the more successful techniques for synthesizing ultrafine ferrites (MFe_2O_3, where M = Co, Ni, Mn, etc.) powder having narrow particle size distribution. The size, shape, and composition of the ferrite nanoparticles very much depend on the type of salts used (e.g. chlorides, sulfates,

nitrates), the M^{2+}/Fe^{3+} ratio, the reaction temperature, the pH value, and ionic strength of the media. This process can avoid complex steps such as refluxing of alkoxides, resulting in less time consumption compared to other techniques [4].

2. Spray pyrolysis:

The principle involved in chemical spray pyrolysis is that when the droplet of spray solution reaches the hot substrate; owing to the pyrolytic decomposition of the solution, well-adherent pinhole-free, uniform films are deposited on the substrate. In this process, the precursor solution is pulverized through the air so that it arrives at the substrates in the form of fine droplets known as aerosols and forms the thin layer at the substrates. The constituents react to form a chemical compound on the substrates. The chemicals are chosen such that the products other than the desired compound are volatile at the temperature of deposition. The composition of the final product is determined by the solute or reactants dissolved in the starting solution in a predetermined stoichiometric ratio [5].

3. Sol-gel synthesis:

Sol-gel technique is one of the most famous solutions processing methods for producing metal oxide nanoparticles. Sol is a colloidal dispersion of the solid particles in a liquid and gel is a state where both liquid and solid are dispersed in each other, presenting a solid network containing liquid

components. In sol-gel processing, a reactive metal precursor, such as metal alkoxide, is hydrolyzed with water, and the hydrolyzed species are allowed to condense with each other to form precipitates of metal oxide nanoparticles. The precipitate is subsequently washed and dried, which is then calcined at an elevated temperature to form crystalline metal oxide nanoparticles [6].

4. **Micro-emulsion:**

A process for the production of metal oxide nanocrystals activated with rare earth elements, which are useful as phosphors, and the nanocrystal oxides are produced by a micellar microemulsion process. In the process, an aqueous solution of the host and activator is prepared and added to a mixture of oil and a micelle forming surfactant and co-surfactant to form the first water in oil microemulsion. An aqueous solution of a hydroxide-containing compound is added to the second mixture of oil and a micelle forming surfactant and co-surfactant to form second water in oil microemulsion. The two microemulsions are added together which causes the coalesce and decoalesce of micelle units to and to form a nanocrystalline hydroxide compound of the host and activator. The solution is washed and treated to remove byproducts. Thereafter, the hydroxide compound is converted to a nanocrystalline-activated oxide [7].

5. **RF plasma torch method:**

In plasma synthesis, the nucleation and growth of particles occur after the gas phase chemical reactions are completed in the plasma plume. The ionized metallic precursors react with oxygen and are cooled rapidly by an inert gas (Ar). Particle nucleation occurs by the condensation of the supersaturated vapor at significant undercooling(provided by the cold inert gas). The critical nuclei approach their final growth forms as the distance from the plasma nozzle exit increases. The growth ceases when the particles travel out of the growth zone to where there is no further supply of atoms, embryos, or nuclei. Finally, the particles are quenched by the cooled reactor walls and collected from the reactor walls [8].

6. **Solvothermal Route:**

Solvothermal (also called hydrothermal) is a synthesis technique for the preparation of magnetic nanoparticles and ultrafine powders. In this method, the reactions are performed in an aqueous media in reactors or autoclaves where the pressure can be higher than 2000 psi and temperatures higher than 200°C. Hydrothermal processing is one of the successful ways to grow crystals of many different materials. This technique has also been used to grow dislocation-free single-crystal particles, and grains formed in this process could have a better crystallinity than those from other processes [9].

7. **Chemical Vapour Deposition:**

Gas phase methods for preparing nanomaterials depend on thermal decomposition (pyrolysis), reduction, hydrolysis, disproportionation, oxidation, or other reactions to cause the precipitation of solid products from the gas phase. In the chemical vapor deposition (CVD) process, a carrier gas stream with precursors is delivered continuously by a gas delivery system to a reaction chamber maintained under a vacuum at a high temperature (>900°C). The CVD reactions take place in the heated reaction chamber and the products combine to form clusters of NPs. Growth and agglomeration of the particles are mitigated *via* the rapid expansion of the two-phase gas stream at the outlet of the reaction chamber [10].

8. Combustion Syntheses:

Combustion syntheses have been applied for the preparation of magnetic nanoparticles. Martirosyan *et al*. produced cobalt ferrite crystalline nanoparticles (50-100 nm) by using the carbon combustion synthesis of oxides (CCSO). In the combustion synthesis process, the exothermic oxidation of carbons generates a thermal reaction wave that propagates through the solid reactants mixture of CoO and Fe_2O_3 converting it to cobalt ferrite. They found that the extensive emission of CO_2 increased the porosity and friability of the product. Also, a complete conversion to ferrite $CoFe_2O_4$ structure was obtained only for carbon concentrations exceeding 12 %wt. Solid-state interactions

between CoO and FeO with the growth of the crystalline cobalt ferrite particles started in the early period of the combustion and continued into the post-combustion zone. As expected, the average particle size increased with increasing combustion temperatures [11].

Noteworthy contribution in the field of proposed work:

1. Z. John Zhang et al (2000) synthesized $CoFe_2O_4$ nanoparticles by using a stable ferric salt of $FeCl_3$ with a micellar microemulsion method. They have found that these $CoFe_2O_4$ nanoparticles have typical superparamagnetic properties. Their superparamagnetic behavior, such as the blocking coercivity and temperature, unambiguously correlates with the particle size. Such correlation is consistent with the Stoner–Wohlfarth theory on single-domain particles [12].

2. Y. Ahn et al (2003) studied superparamagnetic relaxation in cobalt ferrite nanoparticles. These nanoparticles have been synthesized from hydroxide carbonate precursor [13].

3. S. F. Mansour et al (2005) studied frequency and composition dependence on the dielectric properties of Mg-Zn Ferrite [14].

4. Bisht et al (2016), The low reactivity, agglomeration, and oxidation of iron oxide NPs can be avoided by coating them with other metallic oxide NPs, such as zinc nanoparticles.

International status:

Some of the leading institutes outside India that are engaged in research on magnetic nanomaterials are:

1. The influence of milling and subsequent annealing on nickel-ferrite phase formation has been investigated by A. Azizi et al. They have found that the magnetic properties of the milled powder are extensively affected by the annealing temperature [20].
2. Ferrite-polymer nanocomposite spheres from nickel-ferrite or nickel-zinc-ferrite and hydrophilic polymers such as polyhydroxylated methylmethacrylate (PHPMMA) or polyvinyl alcohol (PVA) have been synthesized by S. Sindhu et al. They have confirmed that the dispersion of the magnetic filler occurs in the composite with the help of energy dispersive X-ray diffraction studies and FITR analysis [21].
3. The one-pot reaction of metal acetylacetate with 1, 2-alkanediol in the presence of oleic acid and oleylamine has been investigated by Jin Xie et al. They have discussed that the size of the particles can be tuned by the reactant

concentration and surfactant/metal ratio and MFe_2O_4 nanoparticles with size up to 15 nm via this one-pot reaction process. [22].

4. Gerald F. Dionne et al have demonstrated a new class of radio frequency (RF) devices that combine low-insertion-loss superconductor circuits with variable magnetization ferrites. The devices are designed to prevent penetration of the superconductor by DC magnetic fields associated with the ferrite, thus preserving the low Radio frequency loss of the superconductor while allowing interaction of the Radio Frequency magnetic fields with the magnetized ferrite [23].
5. Co-precipitated Ni-Zn ferrites display a better degree of crystallinity than the Mn-Zn ferrites which are slightly amorphous which have been observed by B. Parvatheeswara Rao et al. They have found that the ball-milled Ni-Zn-In-Ti ferrite samples experience smaller intensities and higher line broadening due to surface disorder introduced during milling [24].
6. Cu-Zn ferrites have been prepared from annular oxides using the standard double sintering ceramic technique by M. A. Ahmed et al. They have discussed the effect of Q-switched Nd: YAG laser irradiation with a wavelength of 1064nm on the electrical properties of the prepared samples Cu_{1-x} $Zn_xFe_2O_4$ ($0.1 \leq x \leq 0.6$) [25].

7. M. Georgescu et al have shown that the magnetization pattern of an assembly of cobalt-ferrite nanoparticles is determined by the competition between collective dipole-dipole interactions and alignment of the dipole moments along the easy axes of magnetization [26].
8. The feasibility of a new concept of realizing on-chip inductors and transformers in power SOCs using existing bond wires with additional ferrite epoxy glob coating has been investigated by Jian Lu et al. They have investigated the concept both experimentally and with finite element modeling [27].
9. Tuning of magnetic properties in cobalt ferrite nanocrystals has been explained by Y. Cedeno-Mattei et al. They have found the results of a synthesis approach in which size control was achieved by modifying the over-saturation conditions during ferrite formation in water through a modified co-precipitation approach [28].
10. The cobalt ferrites with $Co_{0.4}Ni_{0.3}Zn_{0.3}Fe_2O_4$ have been prepared using mechanically alloyed nanoparticles by Samaila Bawa Waje et al. They have found that the permeability spectrum of the resulting ferrite samples shows increasing values with the increasing sintering temperature, this can be attributed to the grain-growth, consequence to sintering [29].

National status:

1. Superparamagnetic particles of substituted ferrites [$Fe_{1-x}B_xFe_2O_4$, B = Mn and Co] and their fatty acid-coated water-based biocompatible ferrofluids have been successfully prepared by Dhirendra Bahadur et al at IIT Bombay with the help of co-precipitation technique using NH_4OH/TMAH as a base. They maintained the stoichiometry of ferrite nanoparticles in this preparation process [30].

2. A room-temperature solution process for the synthesis of Fe_3O_4 nanoparticles has been demonstrated by Joshy Joseph et al at the Department of Physics, Barkatullah University, Bhopal. They have shown that the particle size can be controlled with a low concentration of oleic acid and discussed the structure of as-grown particles using X-ray diffraction and Rietveld refinement [31].

3. The ferrofluid-polymer-composite material has been synthesized by R. P. Pant et al at National Physical Laboratory, New Delhi. They observed that this material has the properties of microwave absorption due to the fast spin relaxation of super-paramagnetic particles distributed in the polymer matrix. This kind of composite film may find good applications in microwave absorption devices [32].

4. PANI/ferrite/alkyd nanocomposite coating based on the nanotechnology approach has been developed by J. Alam et al at the Department of Chemistry,

Jamia Millia Islamia University, New Delhi. They have found that nanocomposite coatings are keenly able to effectively protect steel from electrochemical corrosion in acid and alkaline media [33].

5. The two-phase closed thermosyphon method with nanofluids has been developed by Balkrishna Mehta and Sameer Khandekar at the Department of Mechanical Engineering, IIT Kanpur [34].

6. Samples of nanocrystalline $Ni_xZn_{1-x}Fe_2O_4$ have been successfully synthesized using the chemical co-precipitation method by P. B. C. Rao and S. P. Setty at Andhra University, Visakhapatnam, They have discussed the effect of sintering temperature on the morphological, electrical, and dielectric properties [35].

7. Single-phase cobalt ferrite nanoparticles have been prepared by co-precipitation route followed by annealing at different temperatures by Kinnari Parekh at the Department of Physics, IIT Gandhinagar, Ahmedabad. She has found from the magnetic measurement that coercivity and saturation magnetization increases systematically with an increase in annealing temperature [36].

8. Nickel ferrite nanoparticles have been prepared by S. N. Dolia et al at the Department of Physics, University of Rajasthan, Jaipur. They found the band gap of $NiFe_2O_4$ nano-material is 2.5 eV in pellet form [37].

9. $Mn_{1-x}Zn_xFe_2O_4$ ferrite Nanoparticles have been synthesized using the co-precipitation method by D. Santhosh Kumar et al at Solid State Physics & Materials Science Research Laboratories, Department of Physics, Andhra University, Visakhapatnam. They observed saturation magnetization (M_s), remanent magnetization (M_r), and coercive force (H_c) [38].

Proposed methodology and characterization:

A survey of the literature reveals that global research activities on ferrites nanoparticles are being perused with possible applications in electronics, electricals, optoelectronics, memories, the biomedical field, etc. Most of the reported work has utilized powder precipitation followed by the chemical co-precipitation method. The rich crystal chemistry in spinel ferrite systems offers excellent opportunities for understanding and fine-tuning the super-paramagnetic properties of NPs by chemical manipulations.

The chemical co-precipitation method will be used to synthesize ferrite nanoparticles at different molarities and temperatures by using Ocimum tenuiflorum(Tulsi). We will examine the influence of the growth process variables on the size and structure. Structural, morphological analysis, and

magnetic properties will be investigated using XRD, AFM, TEM, VSM, etc. FT-IR will be used to confirm the attachment of different functional groups in each step of functionalization. A brief description of the characterization strategies to be adopted has been given in the following section:

1. **Structural Analysis:**

X-Ray diffraction technique has been widely used for structural characterization. It depends upon Braggs's diffraction law. This is a reliable technique to find out the crystal structure lattice parameter. Crystalline size can be obtained by the Deby Scherrer formula. In general, the broadening of the diffraction profile can be used to calculate the average particle size and non-uniform strain, while the peak shift can be used for the estimation of uniform microstrain.

2. **Morphological Analysis & Bacterial Studies:**

The morphological analysis will be carried out using AFM, STM, and TEM. STM and AFM are the best surface topographic techniques with atomic-scale resolution. For magnetic materials, many kinds of magnetic interactions are measured by MFM, including magnetic dipole-dipole interaction.

Transmission electron microscopy (TEM) will be used in the determination of particle size. It gives access to a number-weighted mean value.

High-resolution transmission electron microscopy (HR-TEM) gives access to the atomic arrangement. It will be used to study local microstructures (such as lattice vacancies and defects, lattice fringe, glide plane, or screw axes) and the surface atomic arrangement of crystalline nanoparticles.

3. Magnetic Property:

Magnetic Properties will be characterized using VSM and SQUID. The VSM is based upon Faraday's law according to which an e.m.f. is induced by a time-varying magnetic flux in a conductor. In VSM, a sample will be magnetized by a homogenous magnetic field that will be vibrated sinusoidally at a small fixed amplitude with respect to stationary pick-up coils. It is used to evaluate the magnetization of the magnetic nanoparticles as a function of an applied external magnetic field (H) generally between -3 and +3 Tesla. Based on the obtained VSM curve at low and room temperatures, the magnetic behavior of the magnetic nanoparticles can be identified.

SQUID magnetometry is routinely used to assess magnetic properties. Monitoring magnetization as a function of temperature for particles to be cooled with or without an applied magnetic field followed by warming particles in the presence of a magnetic field allows us to determine the characteristic blocking temperature.

4. **Experimental Plant :** Tulsi's scientific name is Ocimum tenuiflorum. In India, tulsi is a household plant that is commonly grown. At various locations, it is also known by a variety of names, including Holy Basil in English, Thulasi in Tamil, Tulasi in Punjabi, and Imli in Urdu, among others. They adore the goddess Tulsi. Tulsi is renowned for its therapeutic benefits, antiviral, and antibacterial characteristics that assist to purify the air. Tulsi-derived medications are used to treat stress, fever, reduce inflammation, and boost stamina. It is an annual shrub that typically grows 2 to 4 feet tall. Little, purple flowers are in bloom. Although it can be found all throughout India, it is most frequently seen in MP.

WORK PLAN:

YEAR – I (15 January 2023 – 14 January 2024)

ACTIVITY	MONTHS											
	1	2	3	4	5	6	7	8	9	10	11	12
Survey of literature and Compilation of review chapter												
Repeated Surveys, Synthesis using CBD, and monitoring of Structural analysis												
Data collection and Reitveld analysis of observed XRD												
Repeat Homogenius sample preparation and data interpretation												

of XRD & TEM												
Data compilation of results, prepare Journal article & International conferences.												
Preparation & Submission of the Final Report progress report												

YEAR – II (15 January 2024 – 14 January 2025)

ACTIVITY	MONTHS											
	1	2	3	4	5	6	7	8	9	10	11	12
Compilation of Introduction, review & experimental technique chapter												
AFM or SEM Study of ZnO –iron oxide core-shell.												
Start writing Paper 2 & 3 on conventional ZnO-Fe2O3 core shell												
Repeat Homogenius sample preparation and data interpretation for the final check, Biological activity & characterized again in IUC –UGC DAE.												
Data compilation of chapter 3 & 4 results & Discussion, International conferences & patents.												
Final Report of progress & thesis preparation.												

EXPECTED OUTCOMES (Nu. 5):

1. Used as Ideal raw materials

2. Reduce energy consumption

3. Product optimization (like Stability and Reusability)

4. Improvement of eco-friendly and reliable processes for the synthesis of metallic nanoparticles is a significant step in the field of applied nanotechnology.

5. Encompasses stability and aggregation of nanoparticles, managing the crystal growth, size, and morphology.

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